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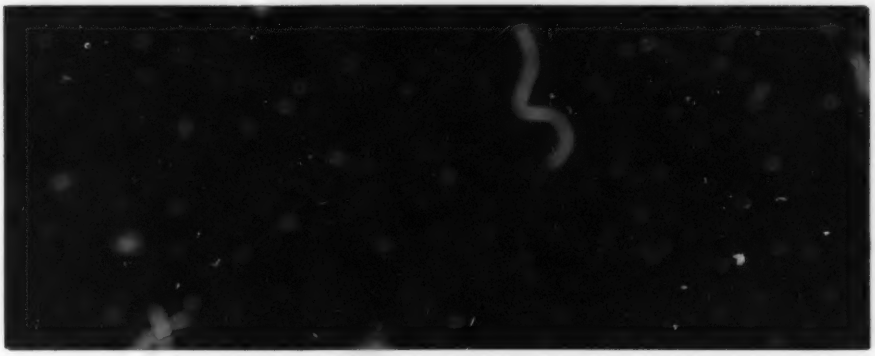

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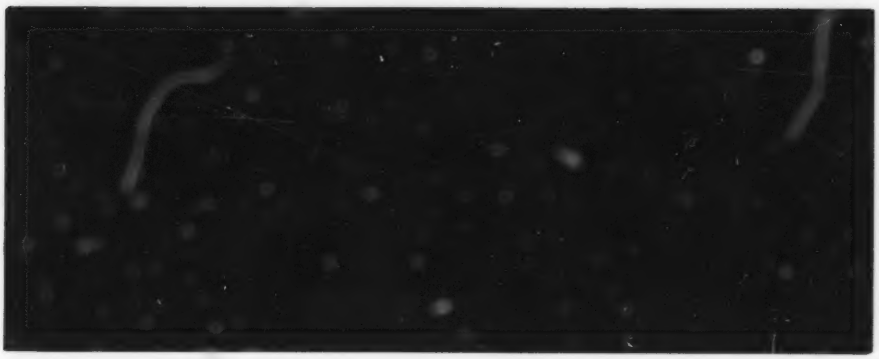

Final Report

R-88-DW-0306

Evaluation of Yield Quality Glyphosate Preharvest

Funded by
The Agriculture Development Fund

92-06747/

K-88-DW-0306

Residue Report to Focus on Inputs for the 1989 Growing Season:

Residues of glyphosate and AMPA in Wheat Seed and Straw, Canola Seed, Barley Seed and Straw, Flax Seed, Field Pea Seed and Straw, and Lentil Seed

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Abstract

Through capital funds from Focus on Inputs, an analytical laboratory for the analysis of glyphosate and its major metabolite AMPA has been set up at the Agriculture Canada Research Station in Regina. An analytical method which employs high pressure liquid chromatography has been adapted which permits quantification of both glyphosate and AMPA at the 0.05 to 0.1 ppm levels in the seed and straw of wheat, barley and field peas, and in the seed of canola, flax and lentils. All six crops were grown for residue analysis in the 1989 growing season. Wheat and canola were grown at three sites in western Canada, whereas flax, field peas, lentils and barley were grown at single sites in western Canada. All samples have been analyzed.

Introduction

Pre-harvest treatment or desiccation of crops has been proposed as a strategy to improve efficiency in harvesting as well as increasing the quality of the seed. In the 1989 growing season, four application rates (0, 0.44, 0.88 and 1.76 kg ha^{-1}) were applied to all six crops at four growth stages (the first stage was at 50% moisture in the seed head/pod and subsequent stages corresponded to 1 week intervals thereafter). Samples for residue analysis were collected from five sites in western Canada (Agriculture Canada Research Stations at Melfort, SK, and Lacombe and Beaverlodge, AB, the Agriculture Canada Experimental Farm at Scott, SK, and ICMS INC., Portage la Prairie, MB). The major objective was to analyze the following for residues of glyphosate and AMPA: (i) canola seed samples and wheat seed and straw samples from a minimum of three of these sites, and (ii) barley and field pea seed and straw samples, and lentil and flax seed samples from one of these sites.

Experimental

All seed samples were milled through a 1-mm screen using a Brinkmann centrifugal mill. A Christy Norris hammer mill was used to mill straw samples. Residues of glyphosate and AMPA were determined using high pressure liquid chromatography and employing fluorescence detection following post-column derivatization. In order to check the efficiency of the methodology, samples fortified with glyphosate and AMPA were also analyzed such that one sample in every twelve samples analyzed was a fortified sample. The details of the analytical methods and sampling procedures will be published elsewhere.

Results and Discussion

Glyphosate and AMPA residue data for the 1989 growing season have been summarized for all crops in Tables 1 - 8 and are uncorrected for recoveries. Wheat seed and straw were analyzed from three sites (Beaverlodge, Portage la Prairie, and Scott), canola seed from three sites (Beaverlodge, Lacombe and Scott), barley seed and straw from one site (Beaverlodge), field pea seed and straw from one site (Beaverlodge), flax seed from one site (Melfort), and lentil seed from one site (Scott). Unfortunately, the lentil seed samples from Scott produced a large interfering peak at the retention time for glyphosate so that residue data for glyphosate are not available for this crop. The problem of this interfering lentil co-extractive did not occur with the lentil seed used during method development.

Observations regarding the residue data for the 1989 growing season are in general agreement with those from the 1988 growing season. The residue data (Tables 1 - 8) for all crops from all sites indicate that the magnitude of both glyphosate and AMPA residues in the seed and straw increased with the application rate; that is, the higher the application rate, the greater the residues in the seed and straw. As well, residues in the seed were dependent upon the stage of the crop at the time of application. The earlier the crop stage at application, the higher the residues of both glyphosate and AMPA observed in the seed.

The principal Agriculture Canada researchers (Kirkland, Harker, Darwent, Townley-Smith and Cessna) will be meeting during the last week in November, 1990 to discuss i) possible environmental and physiological factors affecting residues, ii) relations between residues and crop yield, seed germination etc., and iii) the publication in scientific journals of the residue and agronomic data generated under this project.

Publications:

1. Cessna, A.J. 1988. Annual Report to Focus on Inputs - Residues of glyphosate and AMPA in wheat and canola (presented to 1988 Annual Meeting in Saskatoon, Mar/89).

2. Cessna, A.J.; Etter, M.L.; Caldwell, B.C. 1989. Determination of glyphosate and its major metabolite in wheat seed following pre-harvest treatment at various crop stages. Proceedings of the 24th Annual Workshop for Pesticide and Trace Organic Residue Analysts (Western Canada) pp 87-89. (presented in Winnipeg, Apr/89).

3. Cessna, A.J. 1989. Residue Report to Focus on Inputs for the 1988 Growing Season - Residues of glyphosate and AMPA in wheat seed and straw and in canola seed (forwarded to Focus on Inputs Jan/90).

4. Cessna, A.J. 1990. Annual Report to Focus on Inputs - Complete residue report for the 1988 growing season and partial residue report for the 1989 growing season (presented to the 1989 Annual Meeting at Dana Radar Base, Mar/90).

5. Cessna, A.J. and Etter, M.L. 1990. Determination of glyphosate and its major metabolite AMPA in canola seed following pre-harvest treatment at various crop stages. Proceedings of the 25th Annual Workshop for Pessticide and Trace Organic Residue Analysts (Western Canada) pp 21-29. (presented in Vancouver, Apr/90).

Employees:

Two employees were hired by Focus on Inputs through ADF funding to conduct residue analyses:

Employee	Date Started	Percent of Time Spent on Focus Project	Date Terminated
M.L. Etter	Oct/88	100%	Aug/90
M. Otte	Sep/89	100%	Aug/90

Table 1: Residues (mg kg^{-1}) of glyphosate and AMPA in wheat seed following pre-harvest treatment of wheat with glyphosate using four application rates (0, 0.44, 0.88, 1.76 kg ha^{-1}) at each of four growth stages at three sites in western Canada in 1989.

Application Rate (kg ha^{-1})	Analyte	Analyte Residues (mg kg^{-1})			
		Stage 1	Stage 2	Stage 3	Stage 4
		(Beaverlodge)			
0 (check)	gly	0.03 ± 0.03	0.02 ± 0.01	0.05 ± 0.03	0.01 ± 0.01
	AMPA	0.01 ± 0.01	0.01 ± 0.01	0.01 ± 0.01	ND
0.44	gly	9.59 ± 4.21	0.73 ± 0.34	1.07 ± 1.29	0.62 ± 0.22
	AMPA	0.67 ± 0.27	0.10 ± 0.04	0.05 ± 0.06	ND
0.88	gly	14.8 ± 6.4	4.15 ± 1.72	1.93 ± 0.32	0.55 ± 0.31
	AMPA	1.13 ± 0.59	0.25 ± 0.09	0.09 ± 0.01	ND
1.76	gly	24.5 ± 7.6	13.2 ± 6.4	2.63 ± 1.48	1.61 ± 1.22
	AMPA	2.02 ± 1.28	0.45 ± 0.13	0.12 ± 0.09	0.04 ± 0.05
		(Portage la Prairie)			
0 (check)	gly	0.04 ± 0.03	0.06 ± 0.01	0.05 ± 0.01	0.03 ± 0.02
	AMPA	ND	ND	ND	ND
0.44	gly	0.58 ± 0.20	0.11 ± 0.09	0.04 ± 0.02	0.24 ± 0.12
	AMPA	0.01 ± 0.02	ND	ND	ND
0.88	gly	1.08 ± 0.64	0.12 ± 0.06	0.06 ± 0.03	0.62 ± 0.11
	AMPA	0.06 ± 0.06	ND	ND	ND
1.76	gly	2.23 ± 1.53	0.30 ± 0.25	0.09 ± 0.02	0.85 ± 0.75
	AMPA	0.08 ± 0.07	ND	ND	0.03 ± 0.07
		(Scott)			
0 (check)	gly	0.19 ± 0.14	0.06 ± 0.08	0.01 ± 0.01	ND
	AMPA	0.02 ± 0.02	ND	ND	ND
0.44	gly	7.27 ± 1.18	0.69 ± 0.04	0.05 ± 0.05	0.08 ± 0.02
	AMPA	0.23 ± 0.04	0.02 ± 0.02	ND	ND
0.88	gly	12.5 ± 0.36	1.37 ± 0.78	0.11 ± 0.06	0.12 ± 0.05
	AMPA	0.37 ± 0.04	0.02 ± 0.03	ND	ND
1.76	gly	21.8 ± 2.24	4.28 ± 0.75	0.27 ± 0.21	0.11 ± 0.02
	AMPA	0.70 ± 0.15	0.10 ± 0.02	ND	ND

Table 2: Residues (mg kg^{-1}) of glyphosate and AMPA in wheat straw following pre-harvest treatment of wheat with glyphosate using four application rates (0, 0.44, 0.88, 1.76 kg ha^{-1}) at each of four growth stages at three sites in western Canada in 1989.

Application Rate (kg ha^{-1})	Analyte	Analyte Residues (mg kg^{-1})			
		Stage 1	Stage 2	Stage 3	Stage 4
		(Beaverlodge)			
0 (check)	gly	0.02 ± 0.02	ND	0.03 ± 0.03	0.01 ± 0.02
	AMPA	0.04 ± 0.04	ND	ND	ND
0.44	gly	1.55 ± 0.19	0.93 ± 0.25	2.41 ± 2.62	17.3 ± 1.6
	AMPA	ND	ND	0.21 ± 0.26	0.52 ± 0.08
0.88	gly	4.94 ± 0.68	6.71 ± 2.74	3.94 ± 1.55	13.9 ± 8.4
	AMPA	0.34 ± 0.16	0.21 ± 0.19	0.15 ± 0.06	0.26 ± 0.11
1.76	gly	11.2 ± 8.6	14.0 ± 7.8	8.53 ± 5.42	28.4 ± 9.6
	AMPA	0.53 ± 0.24	0.49 ± 0.13	0.34 ± 0.23	1.08 ± 0.79
		(Portage la Prairie)			
0 (check)	gly	0.17 ± 0.10	0.15 ± 0.14	0.03 ± 0.02	0.02 ± 0.04
	AMPA	0.03 ± 0.03	ND	ND	ND
0.44	gly	0.20 ± 0.10	0.51 ± 0.14	0.16 ± 0.08	0.60 ± 0.13
	AMPA	ND	ND	ND	ND
0.88	gly	0.33 ± 0.21	0.64 ± 0.29	0.42 ± 0.33	1.46 ± 0.32
	AMPA	0.01 ± 0.02	ND	ND	0.14 ± 0.02
1.76	gly	0.67 ± 0.10	2.00 ± 1.00	1.15 ± 0.09	5.86 ± 3.04
	AMPA	ND	0.09 ± 0.08	0.01 ± 0.02	0.98 ± 0.60
		(Scott)			
0 (check)	gly	0.06 ± 0.04	0.22 ± 0.22	0.07 ± 0.06	0.06 ± 0.02
	AMPA	ND	ND	0.01 ± 0.01	ND
0.44	gly	2.14 ± 0.36	2.57 ± 0.31	0.83 ± 0.21	1.00 ± 0.24
	AMPA	0.08 ± 0.02	0.05 ± 0.05	0.06 ± 0.07	0.09 ± 0.02
0.88	gly	3.69 ± 0.49	8.04 ± 2.50	1.59 ± 0.46	1.73 ± 0.20
	AMPA	0.13 ± 0.04	0.18 ± 0.04	0.14 ± 0.02	0.15 ± 0.02
1.76	gly	11.1 ± 2.74	24.3 ± 5.3	2.89 ± 0.23	3.73 ± 0.91
	AMPA	0.27 ± 0.04	0.71 ± 0.20	0.31 ± 0.04	0.33 ± 0.09

Table 3: Residues (mg kg^{-1}) of glyphosate and AMPA in canola seed following pre-harvest treatment of canola with glyphosate using four application rates (0, 0.44, 0.88, 1.76 kg ha^{-1}) at each of four growth stages at three sites in western Canada in 1989.

Application Rate (kg ha^{-1})	Analyte	Analyte Residues (mg kg^{-1})			
		Stage 1	Stage 2	Stage 3	Stage 4
		(Beaverlodge)			
0 (check)	gly	0.69 ± 0.75	0.04 ± 0.05	0.01 ± 0.01	0.03 ± 0.04
	AMPA	0.01 ± 0.02	ND	ND	ND
0.44	gly	22.8 ± 6.7	1.32 ± 0.49	0.27 ± 0.10	0.25 ± 0.09
	AMPA	0.35 ± 0.07	ND	ND	0.01 ± 0.01
0.88	gly	40.5 ± 14.5	1.52 ± 0.60	0.26 ± 0.17	0.46 ± 0.22
	AMPA	0.84 ± 0.22	0.02 ± 0.03	ND	ND
1.76	gly	66.2 ± 15.7	4.99 ± 3.34	0.39 ± 0.12	0.59 ± 0.08
	AMPA	0.83 ± 0.28	0.05 ± 0.04	ND	ND
		(Lacombe)			
0 (check)	gly	0.05 ± 0.06	0.03 ± 0.03	0.03 ± 0.03	ND
	AMPA	ND	ND	ND	ND
0.44	gly	11.2 ± 0.7	3.81 ± 0.66	0.64 ± 0.38	0.54 ± 0.41
	AMPA	0.31 ± 0.01	0.11 ± 0.02	0.01 ± 0.02	0.01 ± 0.02
0.88	gly	16.9 ± 2.9	5.47 ± 3.16	1.12 ± 0.45	0.89 ± 0.33
	AMPA	0.46 ± 0.07	0.10 ± 0.06	0.01 ± 0.01	ND
1.76	gly	33.5 ± 1.2	9.97 ± 4.54	2.23 ± 1.77	0.65 ± 0.16
	AMPA	0.85 ± 0.11	0.16 ± 0.08	0.05 ± 0.05	ND
		(Scott)			
0 (check)	gly	0.11 ± 0.06	0.09 ± 0.06	ND	0.06 ± 0.06
	AMPA	0.01 ± 0.01	ND	ND	ND
0.44	gly	11.4 ± 2.0	1.79 ± 0.22	0.21 ± 0.17	0.18 ± 0.14
	AMPA	0.10 ± 0.02	0.01 ± 0.01	ND	ND
0.88	gly	31.7 ± 3.5	5.82 ± 0.99	0.55 ± 0.15	0.26 ± 0.11
	AMPA	0.31 ± 0.05	0.06 ± 0.02	ND	ND
1.76	gly	83.8 ± 8.9	13.3 ± 1.39	1.24 ± 0.80	0.22 ± 0.04
	AMPA	0.76 ± 0.14	0.13 ± 0.02	0.01 ± 0.01	ND

Table 4: Residues (mg kg^{-1}) of glyphosate and AMPA in barley seed following pre-harvest treatment of barley with glyphosate using four application rates (0, 0.44, 0.88 and 1.76 kg ha^{-1}) at each of four growth stages at one site in western Canada in 1989.

Application Rate (kg ha^{-1})	Analyte	Analyte Residues (mg kg^{-1})			
		Stage 1	Stage 2	Stage 3	Stage 4
			(Beaverlodge)		
0 (check)	gly	0.05 ± 0.03	0.04 ± 0.01	0.03 ± 0.01	0.05 ± 0.05
	AMPA	0.02 ± 0.02	ND	ND	ND
0.44	gly	0.80 ± 0.10	0.73 ± 0.40	0.30 ± 0.15	0.47 ± 0.29
	AMPA	0.02 ± 0.04	0.04 ± 0.03	ND	0.02 ± 0.03
0.88	gly	0.92 ± 0.25	0.91 ± 0.59	0.67 ± 0.19	0.63 ± 0.35
	AMPA	0.07 ± 0.01	0.03 ± 0.03	0.01 ± 0.02	0.04 ± 0.01
1.76	gly	2.92 ± 0.59	1.80 ± 0.85	0.96 ± 0.36	1.03 ± 0.56
	AMPA	0.13 ± 0.03	0.07 ± 0.03	0.03 ± 0.02	0.06 ± 0.01

Table 5: Residues (mg kg^{-1}) of glyphosate and AMPA in barley straw following pre-harvest treatment of barley with glyphosate using four application rates (0, 0.44, 0.88 and 1.76 kg ha^{-1}) at each of four growth stages at one site in western Canada in 1989.

Application Rate (kg ha^{-1})	Analyte	Analyte Residues (mg kg^{-1})			
		Stage 1	Stage 2	Stage 3	Stage 4
			(Beaverlodge)		
0 (check)	gly	0.05 ± 0.02	0.05 ± 0.04	0.05 ± 0.02	0.05 ± 0.04
	AMPA	ND	ND	ND	ND
0.44	gly	2.20 ± 0.44	2.62 ± 1.18	1.61 ± 0.76	2.18 ± 0.61
	AMPA	0.16 ± 0.05	0.11 ± 0.06	0.05 ± 0.04	0.17 ± 0.11
0.88	gly	3.35 ± 0.80	5.16 ± 3.05	4.26 ± 2.30	2.83 ± 2.56
	AMPA	0.23 ± 0.13	0.18 ± 0.11	0.12 ± 0.06	0.08 ± 0.11
1.76	gly	13.6 ± 2.0	8.92 ± 3.23	8.66 ± 4.05	3.85 ± 1.86
	AMPA	0.44 ± 0.12	0.26 ± 0.04	0.21 ± 0.07	0.14 ± 0.09

Table 6: Residues (mg kg^{-1}) of glyphosate and AMPA in field pea seed following pre-harvest treatment of field peas with glyphosate using four application rates (0, 0.44, 0.88 and 1.76 kg ha^{-1}) at each of four growth stages at one site in western Canada in 1989.

Application Rate (kg ha^{-1})	Analyte	Analyte Residues (mg kg^{-1})			
		Stage 1	Stage 2	Stage 3	Stage 4
		(Beaverlodge)			
0 (check)	gly	0.01 ± 0.01	0.01 ± 0.01	0.06 ± 0.06	0.01 ± 0.02
	AMPA	ND	ND	ND	ND
0.44	gly	16.2 ± 5.8	1.43 ± 0.86	0.75 ± 0.26	0.58 ± 0.43
	AMPA	0.22 ± 0.08	0.01 ± 0.02	0.01 ± 0.01	ND
0.88	gly	33.8 ± 6.1	3.92 ± 2.85	1.37 ± 0.60	2.81 ± 2.04
	AMPA	0.33 ± 0.09	0.05 ± 0.02	ND	0.01 ± 0.01
1.76	gly	37.0 ± 3.6	8.94 ± 4.87	1.35 ± 0.58	8.83 ± 5.06
	AMPA	1.05 ± 0.56	0.04 ± 0.04	ND	0.05 ± 0.05

Table 7: Residues (mg kg^{-1}) of glyphosate and AMPA in field pea straw following pre-harvest treatment of field peas with glyphosate using four application rates (0, 0.44, 0.88 and 1.76 kg ha^{-1}) at each of four growth stages at one site in western Canada in 1989.

Application Rate (kg ha^{-1})	Analyte	Analyte Residues (mg kg^{-1})			
		Stage 1	Stage 2	Stage 3	Stage 4
		(Beaverlodge)			
0 (check)	gly	0.03 ± 0.01	0.03 ± 0.01	0.09 ± 0.06	0.02 ± 0.02
	AMPA	0.01 ± 0.01	0.01 ± 0.01	ND	ND
0.44	gly	19.2 ± 1.4	5.20 ± 2.39	11.0 ± 4.0	15.3 ± 7.4
	AMPA	0.31 ± 0.01	0.07 ± 0.05	0.11 ± 0.08	0.18 ± 0.11
0.88	gly	31.0 ± 10.7	14.9 ± 4.7	16.6 ± 2.7	46.8 ± 10.7
	AMPA	0.34 ± 0.15	0.17 ± 0.08	0.20 ± 0.04	0.37 ± 0.10
1.76	gly	26.3 ± 2.1	26.5 ± 11.3	27.4 ± 9.3	40.8 ± 7.4
	AMPA	0.61 ± 0.09	0.24 ± 0.10	0.21 ± 0.09	0.69 ± 0.27

Table 8: Residues (mg kg^{-1}) of glyphosate and AMPA in flax seed following pre-harvest treatment of flax with glyphosate using four application rates (0, 0.44, 0.88 and 1.76 kg ha^{-1}) at each of four growth stages at one site in western Canada in 1989.

Application Rate (kg ha^{-1})	Analyte	Analyte Residues (mg kg^{-1})			
		Stage 1	Stage 2	Stage 3	Stage 4
			(Melfort)		
0 (check)	gly	0.12 ± 0.04	0.28 ± 0.16	0.15 ± 0.12	-*
	AMPA	0.04 ± 0.06	0.02 ± 0.03	0.03 ± 0.04	-
0.44	gly	1.57 ± 1.01	0.38 ± 0.12	0.32 ± 0.04	-
	AMPA	0.02 ± 0.02	ND	ND	-
0.88	gly	3.08 ± 2.26	1.28 ± 0.41	0.84 ± 0.31	-
	AMPA	ND	0.02 ± 0.01	0.01 ± 0.02	-
1.76	gly	10.6 ± 5.6	1.74 ± 0.38	1.32 ± 0.29	-
	AMPA	0.10 ± 0.11	0.03 ± 0.02	0.01 ± 0.01	-

*Because of drought conditions at this site, the crop was naturally desiccated and the glyphosate treatments were not applied.